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Effect of Heat Treatment on the Microstructure and Mechanical Properties of a Welded AISI 410 Martensitic Stainless Steel

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Abstract: Although martensitic stainless steel materials are not used in large quantities compared to austenitic and ferritic grades, they play a huge and often unseen part in our modern world due to their combination of strength, toughness and moderate corrosion resistance. However, after welding the martensitic stainless steel tend to lose their mechanical/microstructural integrity. In this study, the microstructures and mechanical properties of a welded AISI 410 martensitic stainless steel after different heat treatments were studied, with aims to restore the hardness and improve grain refinement of the materials. The results show that the structures of the steel after austenitizing treatment at 1020°C are of lath martensite mixed with a small amount of retained austenite. Apart from TP2 specimen, where martensite phase was transformed into ferrite structure, the structures of the tempered steel are mixtures of tempered martensite, carbides and reversed austenite dispersed in the martensite matrix. The result indicated that the tempering regimens (500, 600 and 700°C) carried out improved the hardness and grain refinement leading to the existence of finely distributed carbides in the materials. TheTP3 specimen experienced secondary hardening phenomenon, displays the best comprehensive mechanical properties and has the highest hardness value of 370.7 HV close to the parent metal after tempering at 700 °C.

Keywords: Martensitic stainless steel, heat treatment, microstructure, mechanical properties, austenitizing, tempering, welding.

1. INTRODUCTION

Martensitic stainless steels grades containing 12-17% Cr (mass %), containing with sufficient carbon (0.15-1.0 wt % C), has established itself as a competitive class of industrial materials owing to the unique combination of good properties, such as , (1) excellent Corrosion resistance; Corrosion resistance is by far the most important property of a stainless steel. Martensitic stainless steels are usually well resistant to high-temperatures oxidation and localized corrosion, e.g. pitting and crevice corrosions, and stress-corrosion cracking in seawater. Previous reports suggest that martensitic grades are more resistant to stress cracking corrosion (SCC) in chlorides and caustic alkali media than austenitic grades [1]. (2) martensitic stainless steel grades are relative cheaper when compared to the austenitic stainless steel that dominates the world market. The price of Nickel raw materials represent as much as 40–45% of the price of the austenitic stainless steels, such as Type 304. The price of nickel is increasing rapidly due to high demand outstripping its supply, therefore the austenitic stainless steel, which is the largest consumer of nickel, has been maintaining a high-price [2, 3], (3) Good electrical and machinability properties; martensitic stainless steel alloys are increasingly been used in replacing plain steels and plastics due to their excellent reproducibility, electromagnetic properties, and environmental concerns. These qualities made martensitic stainless steel very important and most favoured to replace in part austenitic grades. Currently, the uses of martensitic stainless steel grades in several industrial applications have increased tremendously. Some of its applications include, razor strip, blades and cutting tools, surgical instruments, gears, valves, pumps, shafts, offshore oil and gas components, bearings, mixers and stirrers, turbine parts and aerospace. Many of these applications are hidden to most of us which probably explains why martensitic stainless steels do not have a prominent public profile. It is good to remind ourselves that much of our modern world rests on martensitic stainless steels quietly doing their job behind the scenes [4].

Despite these, martensitic stainless steels with higher carbon contents are susceptible to underbead cracking, especially when hydrogen is present during welding [5]. Because of the internal stresses induced by the volume increase associated with the austenite-to-martensite transformation, underbead cracking can still occur even if relatively low restraint is employed during welding. As a result, martensitic stainless steels with carbon content above 0.25–0.30% are not normally welded. To avoid underbead cracking in martensitic stainless steels, both preheating and post weld tempering (between 600 and 850°C) are usually employed [5]. Austenitic or ferritic-austenitic electrodes are used for welding martensitic stainless steels [6]. This is because the weld metal so produced remains ductile, thus reducing the chance of underbead cracking. The typical heat-treatment sequence for martensitic stainless steels includes annealing to soften the steel in preparation for subsequent cold work or

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machining, austenitizing to form an austenitic structure and fully or partially dissolve carbides, cooling or quenching to transform the austenite to martensite, followed by tempering of the martensitic structure to improve toughness and ductility [7].

According to several authors, [8, 9], the microstructure of martensitic stainless steel consists of martensite, un dissolved carbide as well as retained austenite and the amount of carbide in the as-quenched microstructure influences the properties of this material such as hardness, strength, toughness and wears. The austenitizing temperature employed during heat treatment determines the partitioning of carbon and alloying elements between the austenite and carbide phases, with an increase in temperature leading to increased carbide dissolution, higher dissolved alloying element contents, and unwanted grain growth [7]. Pickering [10] noted that when in solid solution at temperatures above the carbide dissolution temperature, carbon and carbide-forming elements affect the transformation to martensite by depressing the martensite transformation range and reducing the martensite start (M_s) and martensite finish (M_f) temperatures. If the M_f temperature is depressed below room temperature or even below 0^oC, the retained austenite may present in the as-quenched microstructure.

Martensitic stainless steels are finding increasing application as high strength and increased hardness steel in a wide range of applications. Welding is the main manufacturing and maintenance process used.

However, little information on the metallurgical alterations caused by welding of this type of stainless steel is found in the literature, prompting this study. Here, we study the effect of austenitizing and tempering treatments at different temperatures cycles on the microstructures and mechanical properties of the heat affected zone (HAZ) of a welded AISI 410 martensitic stainless steel.

2. MATERIALS AND METHOD

2.1. Specimens and Experimental Set-up

The laboratory samples were machined directly from the bulk AISI 410 martensitic stainless steel supplied by Iron and Steel Cooperative market, Lagos, Nigeria. The nominal chemical composition of the steel used in this work was analysed using Spark Optical Emission Spectrometer model ARL Quanto Desk rating 350VA and listed in Table1.The laboratory specialness were sectioned into rectangular samples of 120 × 20 × 1.2 mm^3 before welding with Electric Arc welding process using E308-16 ISO9001 Certified welding electrodes.

Table 1: Chemical Composition of AISI 410 Martensitic Stainless Steel (wt. %)

	Fe	**C**	**Si**	**Mn**	**P**	**S**	**Cr**	**Ni**	**Mo**
Avg	72.34± 0.0592	0.1597± 0.0073	0.3677± 0.0079	9.935± 0.0746	0.0183± 0.0020	0.0095± 0.0015	13.33± 0.0453	1.083± 0.0153	0.0344± 0.0086
	Cu	**Ti**	**Nb**	**Co**	**V**	**W**			
Avg	1.254± 0.0093	0.000± 0.0000	0.5166± 0.0141	0.1408± 0.0080	0.0910± 0.0036	0.6169± 0.0337			

2.2. Outline of the Experimental Routes for the Heat Treatment Regimes

All specimens were pre-processed by an identical austenitization and cooling route in order to provide a homogeneous ferrite–austenite microstructure before the application of the different tempering treatments. The specimens were pre heated at 250°C in a muffle furnace (Chesterfield, U.K) model LF3 according to the prescription of American Iron and Steel Institute [11] procedures for martensitic grades. After this, the welded specimens were austenitized in a muffle furnace at1020°C, soaked for 30 minutes and oil quenched. To generate more test materials for the required test regimes, the quenched specimens were further sectioned to dimensions 40 mm x 20 mm x 1.2 mm before tempering. The specimens were then tempered at 500°C, 600°C and 700°C in a muffle furnace, soaked for one (1) hour and air-cooled. The codes AR, AW, AS, TP1, TP2 and TP3 were used to denote the as-received, as-welded, austenitized, tempered at 500°C, 600° and 700°C temperatures respectively. The heat treatment cycles used are illustrated in Fig.1.

2.3. Characterization of the microstructure

2.3.1. Light optical microscopy

The specimens for light optical microscopy were mechanically polished to a 1 µm finish and etched using a vilella's solution consisting of 1 gram of picric acid, 10 mL of hydrochloric acid (HCl) and 100 mL of ethanol.Micrographs were taken at a sample location at the joint as well as the HAZ areas to reveal the general microstructure.

2.4. Measurement of the mechanical properties

2.4.1. Hardness testing

The hardness values of the samples were determined using Vickers indenter with a load of 980.7mN and 10 seconds loading time. Several measurements were taken for each experimental condition on the heat affected zone (HAZ) and average values recorded. All of the procedures were carried out according to the ASTM E92-82/E2 standard method [12].

2.5. Grain Size Measurement

Actual average grain size diameter was determined using grain intercept count. This offers clear advantages to the ASTM grain-size number that in reality does not offer any direct information about the actual size of the grain [13]. This was achieved by drawing a random line of 124 mm length on the photomicrograph. The number of grains intersected by this line is then determined, and the ratio of

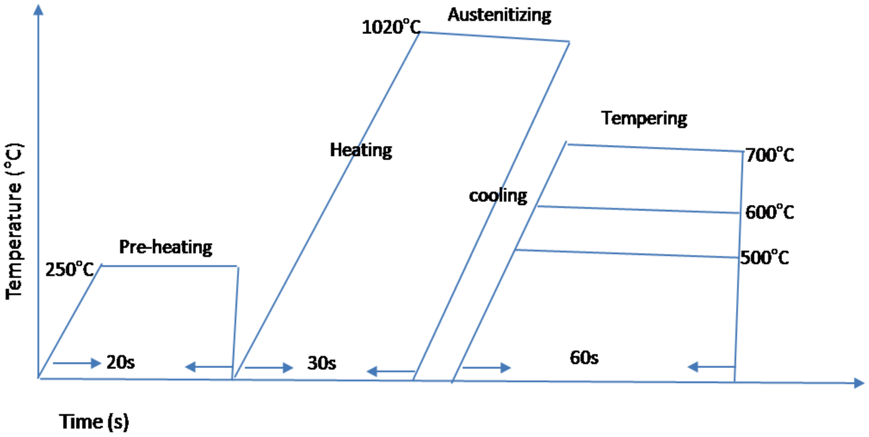

Figure1: An Illustration of the Heat Treatment Regimens Carried out in this Study.

the number of grains to the actual length of the line is determined, n_L. The average grain diameter d is determined using equation one (1) [13].

$$d=C/(n_L.M)\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots(1)$$

Where:
C is a constant (C=1.5 for typical microstructures)
M is magnification at which the photomicrograph is taken.

3. RESULTS AND DISCUSSION

3.1 Microstructural Analysis of the As-Received Sample

Figure 1a is the microstructure of the as-received sample. As shown and pointed out with a red arrows, the microstructure consist of a dual phase of different un dissolved carbide in full martensitic matrix and retained austenite phase. The presence of carbides in martensitic stainless steel microstructure is not surprising because of the high carbon content. It has been established over the years that steels with more than 0.2% carbon and 12-13% chromium content consist of different volumes of M_3C, M_7C_3, and $M_{23}C_6$ carbide precipitate [14]. The precipitation of the carbides is reported to be dependent on time with M_3C precipitating first, followed by M_7C_3, and then $M_{23}C_6$. However, in this investigation, only $M_{23}C_6$ carbides can be said to be identified due to the austenitizing temperature used, with M consisting mainly of iron and chromium. This type of carbide are known to dissolve in the 950-1050°C [15], therefore increasing the volume fraction of austenite in the matrix of the material/sample. From this perspective, we conducted different heat treatment in order to study the effects on the carbides. The results of the study are presented below.

3.2 Effect of Welding on the Microstructure.

After the welding, three zones with different microstructures were observed from the surface towards the center of the specimens: a martensitic surface layer, a transition zone and a dual-phase martensite-ferrite inner zone. The microstructures of the heat affected zones are presented in Fig.1b_1 and 1b_2. As seen in the microstructure of Fig. 1b_1, the point of welding is evident.

The microstructural investigation indicates that it consists of a needle-like plate of martensite, austenite and some carbides.

For the HAZ microstructure shown in Fig. 1b_2, the analysis of the aggregates shows the presence of austenite and ferrite at the grain boundaries while inside the grains contains austenite with different precipitates. We attribute their presence to the increased diffusion of high carbon at high temperature during the welding process.

3.3 Effect Austenitizing Temperature on the Micro structure.

After welding, austenitizing heat treatment was done to increase the volume of austenite in the weld surface. The microstructure shown in Fig. 1c is as a result of this treatment. The microstructure consists of undissolved carbide with retained austenite in a matrix of un-tempered martensite. The result is in good agreement with the work performed by various authors [16, 17]. However, the shape and size of the martensite formed in the HAZ is unchanged after this treatment. This, prompting further heat treatment procedures.

3.4 Effect of tempering temperatures on the Microstructure

After austenitizing, a high fraction of carbides, chromium and other alloying elements are dissolved in the austenite solid solution, thereby making the steel hard and brittle. Tempering treatment was carried out at; i. Obtaining a more appropriate combination of strength and toughness.

ii. To alter phase composition under the influence of tempering treatment. Some of the experimental characteristics are explained below. The micro structural analysis of the sample tempered at 500°C is displayed in Fig.1d.The micrograph of sample revealed retained austenite within the tempered martensite and carbide precipitate. The result is consistent with Sudsakan et al. [18] investigation.

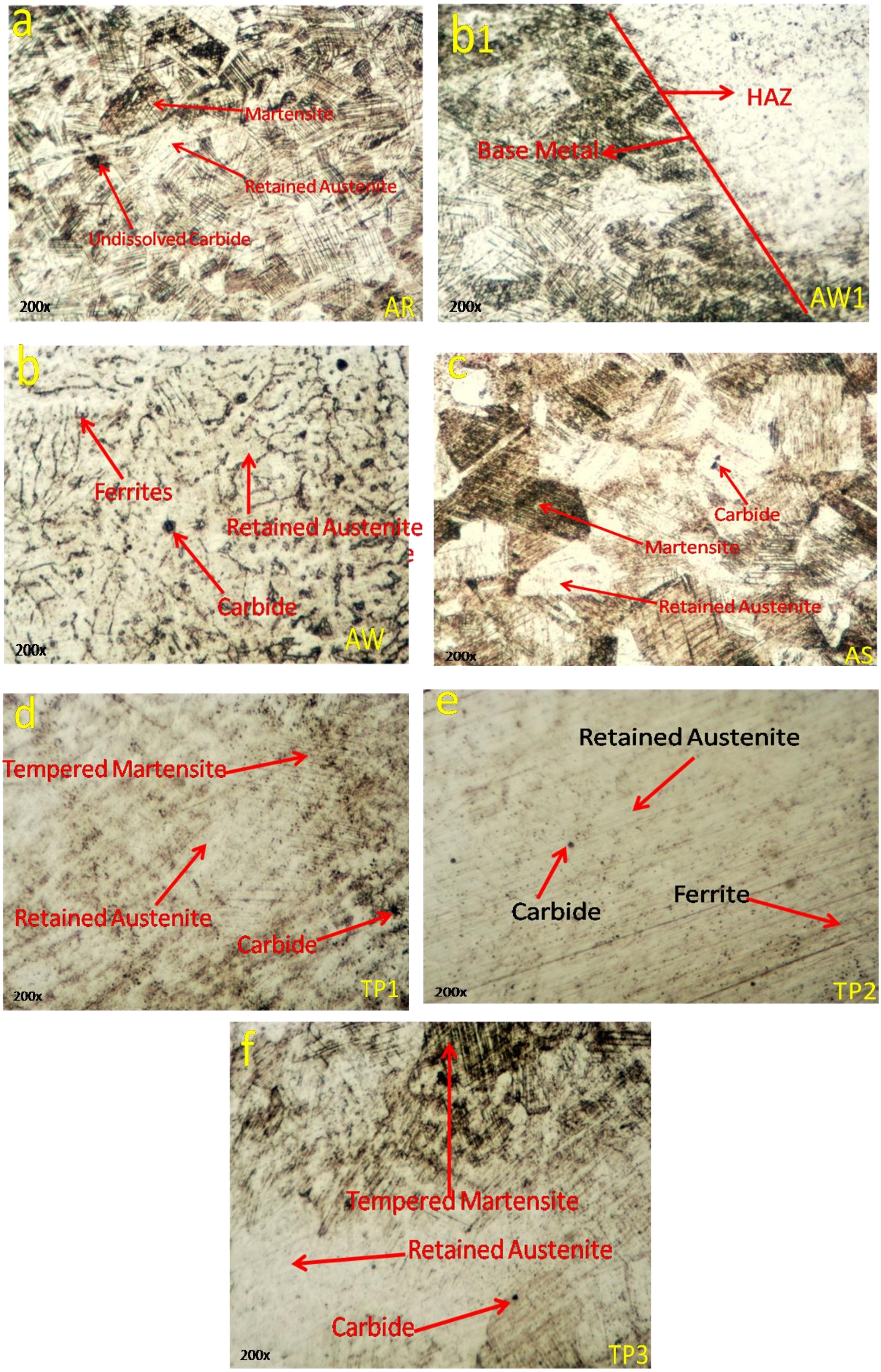

Figure 2: Optical micrograph of the specimens (a) AR, (b1) AW1, (b) AW, (c) AS, (d) TP1, (e) TP2 and (f) TP3

Fig. 1e shows the microstructure of sample tempered at 600° C. As observed from the microstructure, majority of the martensitic structures has been transformed to ferrite and other carbides (indicated by the red arrow). This is in line with previous reports by several authors [19, 20], which shows that at high temperature, i.e.,600°C, the diffusion of the substitutional alloying elements becomes possible, leading to the occurrence of special carbides accompanied by cementite dissolution. This phase transformation is accompanied by the microstructural changes in martensite and ferrite. It is also noticeable that the dislocation density decreased while the sub grains form inside the ferrite grains. Hence, tempering at this temperature initiate a subgrain growth and eventual re-crystallization of grains. Microstructure of the sample tempered at 600°C revealed retained austenite, ferrite phase, and carbide precipitates.

Further heating or tempering at 700°C lead to the refinement of the deformed grains as can be seen in the micrograph. These structures made the ferrite grains look equiaxed. However, it appeared that at high temperature, the ε-carbides has dissolved, while the thin Widmannstatten plate of cementite and an element of tempered martensite occured inside the ferrite grains. The microstructural analysis of the sample tempered at 700°C is displayed in Fig. 1f.

3.5 Effect of Heat Treatment on the Hardness.

Fig. 3 clearly showed that different heat treatment procedures significantly affect the hardness of a welded AISI 410 martensitic stainless steel. The hardness value of 405.7HV was obtained for as- received (AR) sample. The high hardness value of the as-received sample confirms the full martensitic microstructure observed. This can be attributed to the hard martensitic matrix and to the presence of a large concentration of primary carbide.

The measured hardness value for the as-welded (AW) sample drops substantially to 273.2HV. The hardness value decreased to 235.0HV after austenitizing at a temperature of 1020°C. This can be attributed to the homogenous distribution of plate martensite in its microstructure. The hardness decreases and increases again during tempering in the range 500-700°C. This is due to precipitation of carbide when the austenite transformed to martensite during tempering. Measured hardness value for the sample tempered at 500°C (TP1) increases considerably to 333.95HV. The high hardness value can be attributed to the gradual dissolution of chromium carbides which increases the carbon and alloying element contents of the austenite.

The lowest hardness value of 221.7 HV was obtained at a tempering temperature of 600°C (TP2). This confirms the absence of martensite and increased retained austenite which softens the sample. The softening occurred when the M_7C_3 carbides start to coarsen and martensite less tetragonal [21, 22, 23].

The highest heat treated hardness value of 370.7HV obtained was at the tempering temperature of 700°C (TP3) as shown in Fig.3. This is attributed to the transformation of ferrite phase at tempering temperature of 600°C to martensite phase at tempering temperature of 700°C. The high hardness value obtained can also be attributed to the fine martensitic matrix and low levels of retained austenite as well as the presence of a higher amount of carbide, which hardens the matrix when it dissolves. Bhadeshia [24] noted that the recovery strength occurs due to carbide precipitate in extremely fine but dense during tempering at elevated temperature and time. It can be concluded that secondary hardening phenomenon occurred at 700°C tempering temperature.

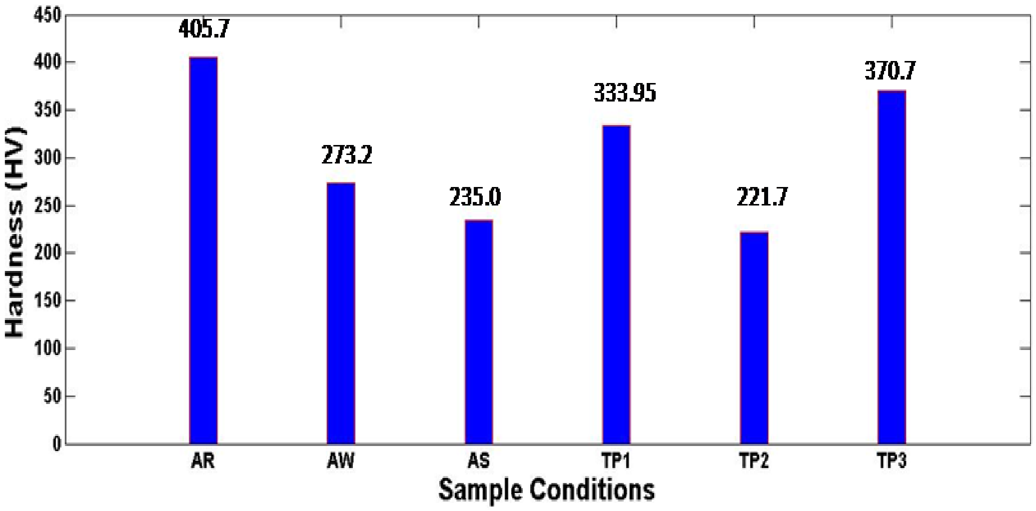

Figure 3: Chart of Hardness Values against Sample Conditions

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3.6 Effect of Heat treatment on the Grain Size.

Grain refinement and material processing are interwoven as microstructural parameters; grain size, grain shape, texture or preferred crystallographic orientation inherently changes with processing conditions [25]. Fig. 4 summarizes the effect of different heat treatment procedures on the specimen's condition.

The smallest grain size obtained at a tempering temperature of 700^{o}C confirmed the hardening effect exhibited by this material. However, the results show that the effect is an inverse on the hardness of the specimens. Hence, study of the relationship between the grain size and hardness becomes imperative.

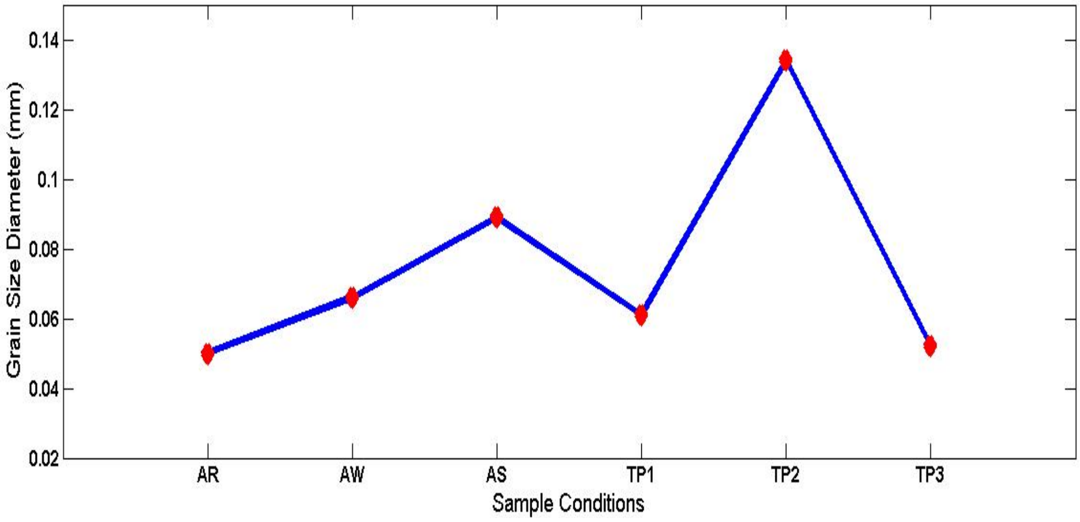

Figure 4: Graphical Representation of Grain Size against Sample Conditions

3.7 Relationship between Hardness and Grain Size

Average grain size is the key micro structural parameter that affects all aspects of the physical and mechanical behaviour of polycrystalline metals, including their chemical and biochemical response to the surrounding media [26]. Fig. 5 shows the relationship between the hardness and grain size and the results appear to closely follow the Hall–Petch relationship. That is, increase in hardness versus decreasing recrystallized grain size. This indicates that the hardness of the AISI 410 martensitic stainless steel is in fact dependent on the actual grain size diameter. The result obtained is in line with previous reports by Hanaruma et al. [27].

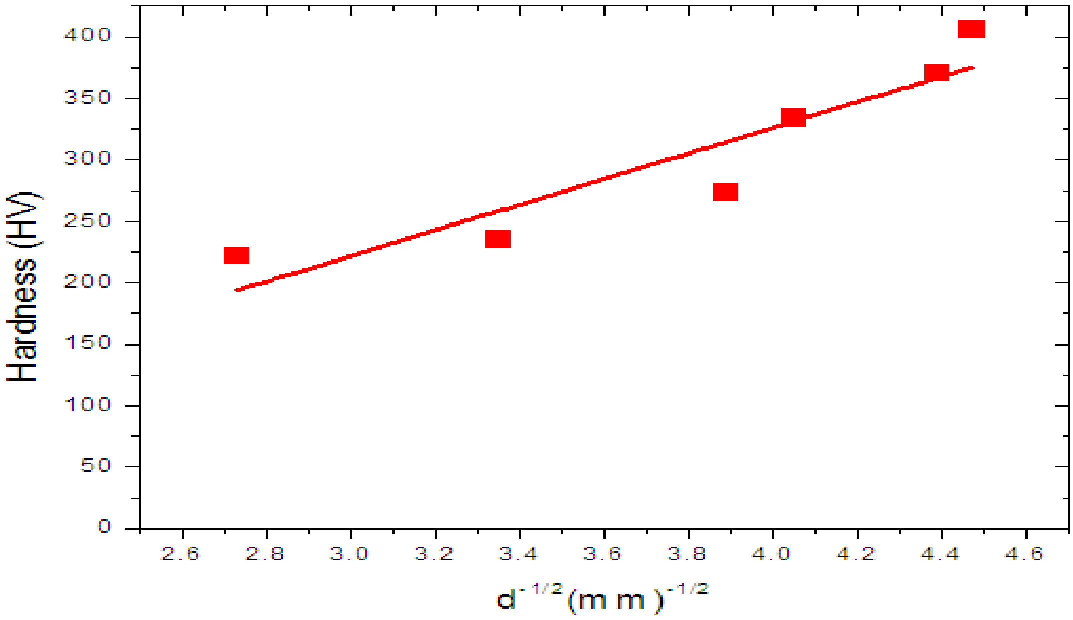

Figure 5: The Relationship between Hardness and Grain size Diameter

4. CONCLUSION

The effect of different heat treatment procedures on the microstructure and mechanical properties of AISI 410 martensitic stainless steel joint welded with E308-16 ISO9001 certified electrodes have been investigated. The conclusions drawn from this study are as follows:

❖ The HAZ of as-welded specimen revealed the presence of austenite and ferrite at grain boundaries while inside the grains contains austenite with different precipitates.

❖ Tempering at 700 ^{o} C for 1hr gives the best heat treatment process for minimal retained austenite, evenly dispersed carbide, high hardness and smallest grain size.

❖ Results of the tempered samples indicated the presence of retained austenite, carbides in tempered martensite matrix except TP2 where martensite phase was transformed into ferrite structure.

❖ Secondary hardening phenomenon was observed at 700^{o}C tempering temperature.

❖ Hall-Petch relation was established using the hardness and grain size variation. That is, HV versus d $^{-1/2}$ curve synchronizes well.

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